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Synthesis, Characterization and Antibacterial Studies of Copper Sulphide Nanoparticles by Chemical Precipation Method

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Abstract

In this present study the non toxic CuS nanoparticles was synthesized by the reaction of copper acetate, thiourea along with the precipitating agent NaoH under chemical precipitation method. The final product CuS nanomaterial was dried at room temperature for better growth of nanoparticles. The size and growth of the crystal depends on the temperature also on the addition of reagent. The resultant nanocrystal were characterized using various techniques like X ray diffraction reveals the particle size, Scanning electron microscope determines the morphology of crystal, Energy dispersive X ray spectroscopy investigate the elemental composition of nanoparticles, U-Visible spectroscopy examine the presence of metallic ion, Fourier transform infrared spectroscopy inspect the existence of functional group. The antibacterial activity of hexagonal structured copper sulphide nanomaterials against gram positive and gram negative bacteria were also analyzed for their wide applications.

Keywords: CuS nanoparticles, Chemical precipitation. Antibacterial activity

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1. Introduction

Nanotechnology has playing a vital role in various industrial and medicinal applications. The particle which is of smaller size ranging from 1nm to 100nm is denoted as nanoparticles.[1] The nanomaterials exhibits physical and chemical property in which the p type semiconductor[2] copper sulphide nanoparticles exposes wide potential application in solar cells, ion storage, lithium ion batteries. In case of semiconductor based application CuS NPs provides an excellent platform in the treatment of cancer. The CuS nanoparticle is composed of copper and sulfur components which have high electrical conductivity and it is a mineral that occurs in nature named as covellit which kills bacteria, exhibit low toxicity and maintain good haemocompatibility. Notably, CuS NPs not only cure infection but also disinfect the pathogens. Various techniques were used for preparation of nanoparticles like sol gel method, hydrothermal method, Co-precipitation method, solvothermal method, electro deposition method etc., The chemical precipitation method is employed for the removal of metals and other inorganics, fats, oils, organic substance from water. Generally, precipitation is method of making the contaminants that are dissolved to settle out of solution, as a solid precipitate which can be filtered, centrifuged or separated from liquid portion. Chemical precipitation is a simple technique which is given more priority due to its low cost, few remains after calcination and easy industrialization.[3-7] Hence this method is employed in this study for obtaining CuS nanoparticles of nanometer size.

2. Experimental procedure

The components of copper and sulphur were taken as Copper acetate and thiourea. Sodium hydroxide pellets were used as a precipitating agent. The chemicals used in this work were analytically pure. In order to prepare CuS nanoparticles, 9.98 gm of copper acetate was dissolved in 250 ml of distilled water and 7.624 gm of thiourea was dissolved in 50 ml of distilled water, 10 gm of sodium hydroxide pellets was dissolved in 20 ml of distilled water. The copper acetate solution was stirred in a magnetic stirrer with constant stirring and thiourea solution is also stirred in a separate stirrer for 10 mins to make a salt to dissolve. The thiourea solution is allowed to mix with the blue coloured copper acetate solution such that the solution turns green

on adding. Then the NaoH was added drop wise in which the solution turns light brown on adding first drop and the colour gets darkened on adding more NaoH. The solution was taken out from the stirrer and it is kept without disturbance for 4 hours such that the precipitate gets settle down. Now the precipitate was filtered and washed using acetone and distilled water to remove impurities and then dried in room temperature for 24 hours and it was kept in oven at 150˚c for 1 hour. Later, kept in furnace at 200˚C for 2hrs to obtain fine nanoparticles.

3. Results and Discussions

3.1. X-ray diffraction studies

The X-ray diffraction pattern of synthesized copper sulphide nanoparticles is compared for different temperatures and is shown in Fig. 1. The pattern consist of 2θ at 29.40˚, 32.00˚, 48.01˚ at room temperature, 32.23˚, 48.06˚, 29.50˚ at 120˚C 32.02˚, 46.50˚, 27.70˚ at 200˚C.[8, 9] From the above comparison it is concluded that the intensity and crystallite size of the crystal increases with increase in temperature and also the growth of crystal completely depends on time period of leaving the sample in room temperature. The full width half maximum values get decreased when the temperature gets increased. The result indicates the improvement of crystalline size of CuS NPs lay on room temperature and annealing temperatures. The crystallite size of CuS NPs is ranging about 3-6nm respectively.

3.2. Scanning Electron Microscope Analysis

The morphology of copper sulphide nanoparticles was examined using scanning electron microscope. The SEM images of CuS nanocrystals exhibits ball like structures[9] with less agglomeration due to smaller amount of impurities in it. The SEM image of copper sulphide particle was taken using different magnifications and it is shown in Figure 2.

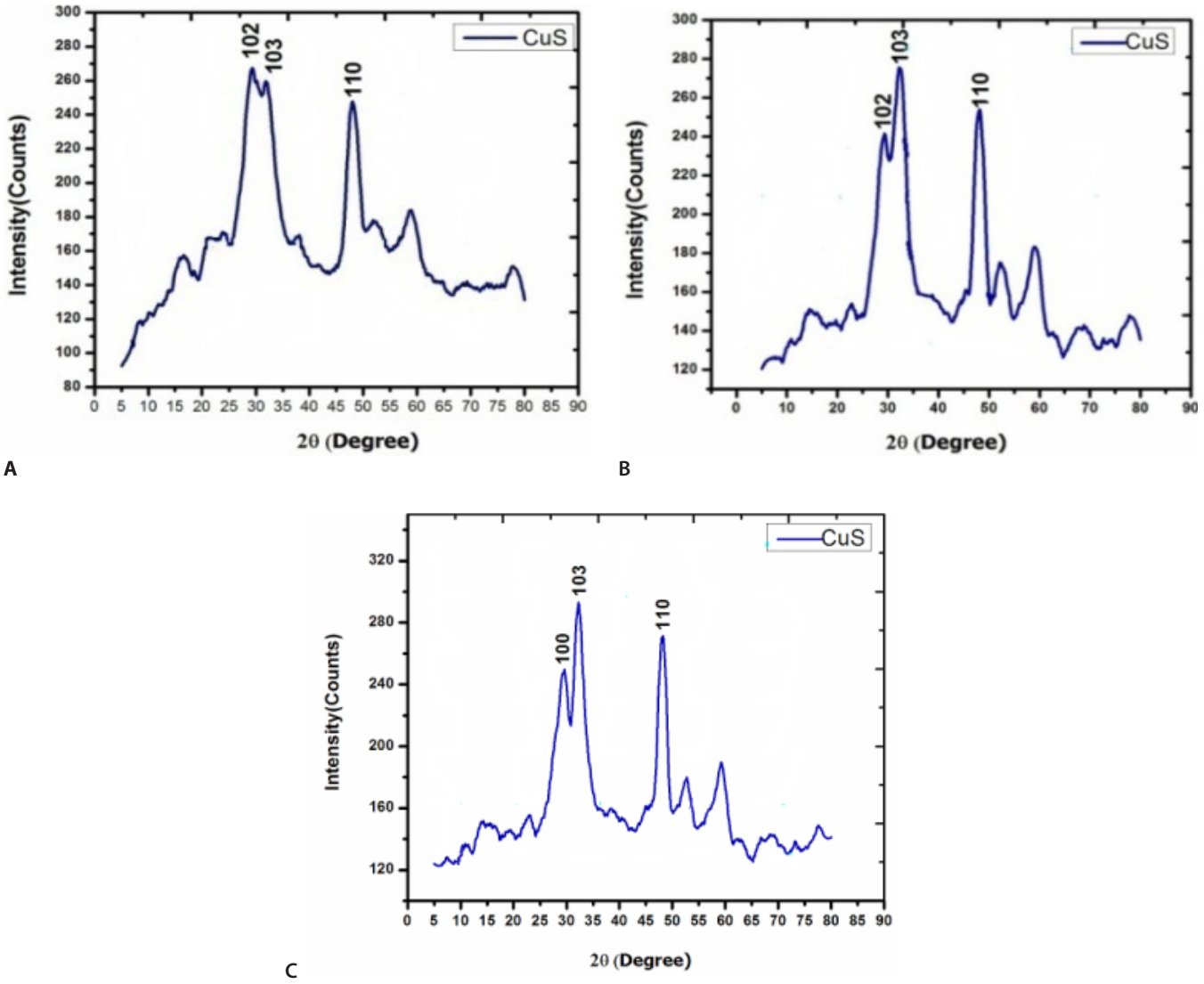

Fig. 1(A to C): XRD pattern for CuS nanoparticles; (A) At room temperature; (B)Calcination at 120˚C; (C Calcination at 200˚C

Table 1: Crystallographic Parameters of CuS Manoparticles

S. No	Sample name	Temperature (˚C)	FWHM (deg)	Average size (nm)
1	CuS1	Room temperature	3.8000	3.096
			3.4000	
			2.0220	
2	CuS2	120˚	1.8714	4.982
			1.4345	
			2.1500	
3	CuS3	200˚	1.5104	5.903
			1.4572	
			1.4334	

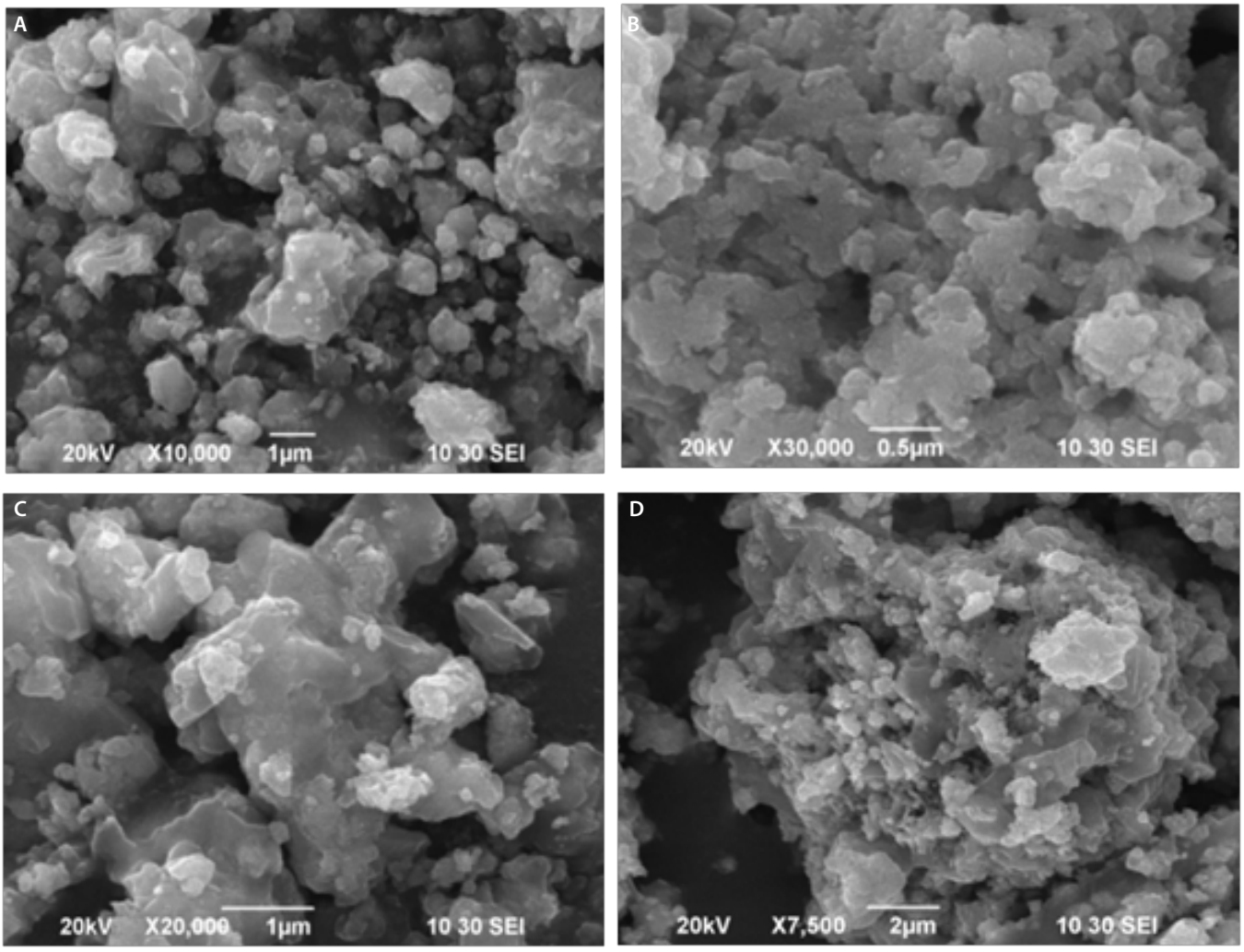

Fig. 2(A to D): SEM images of CuS nanoparticles

3.3 Energy dispersive X- ray

The elemental composition of copper sulphide nanoparticles reveals the presence of Cu, S and O. EDAX spectrum of copper acetate+thiourea is shown in figure that confirms the presence of copper sulphide nanoparticles.

3.4 UV-Visible spectroscopy analysis

The metal ions stability and optical property of copper sulphide nanoparticles was monitored by UV-Visible spectrophotometer. The characterization of nanoparticles was taken out from the range of 190-1100m and the graph

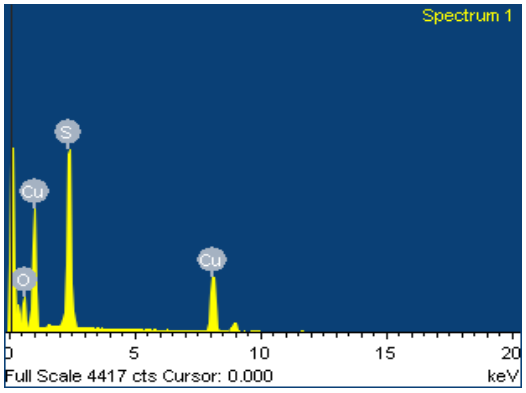

Fig. 3: EDAX Analysis of CuS nanoparticles

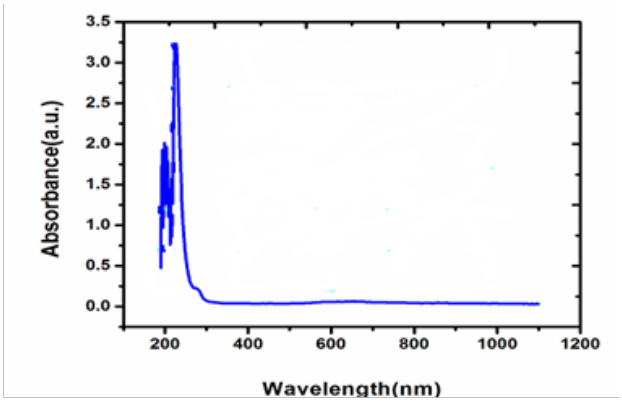

Fig. 4: Optical Analysis of CuS nanoparticles

is plotted between wavelength and absorbance. The UV absorption spectrum and the peak of copper sulphide nanoparticles was obtained at 227-243nm shown in figure. The band gap energy (Eg) was calculated as 5.6 eV.

3.5 Fourier Transform Infrared Spectral Analysis:

FTIR identifies the functional groups of the synthesized sample. The wavelength region was recorded in the range of 400-4000cm^{-1}. The spectrum of CuS nanoparticles acquires the main characteristics peaks at 3429.43 cm^{-1}, 1658.78 cm^{-1}, 603.72 cm^{-1},[7] 1101.55 cm^{-1},[8] 2999.31 cm^{-1}, 1421.54 cm^{-1} .The presence of functional group and wave number was shown in Table 2.

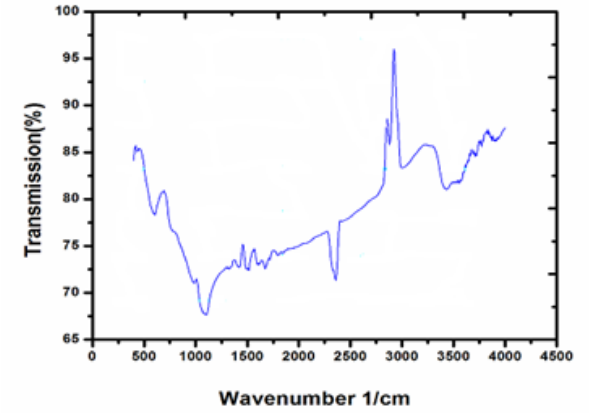

Fig. 5: FTIR spectrum of CuS nanoparticles

Table 2: FTIR data of CuS nanoparticles

Types of Vibration	Wave Number
O-H stretching	3429.43 cm^{-1}
C-H stretching	2999.31 cm^{-1}
C=C stretching	1658.78 cm^{-1}
O-H bending	1421.54 cm^{-1}
C-O stretching	1101.55 cm^{-1}
C-Br stretching	603.72 cm^{-1}

3.6. Antibacterial Activity:

The given sample was tested for antimicrobial activity by well diffusion method. Liquid Mueller Hinton agar media and the Petri plates were sterilized by autoclaving at 121° C for about 30 minutes at 15 lbs pressure. Under aseptic conditions in the laminar airflow chamber, about 20ml of the agar medium was dispensed into each Petri plate to yield a uniform depth of 4mm. After solidification of the media, 18 hrs culture of Gram positive microorganisms such as Bacillus cereus(MTCC 430), Staphylococcus aureus (MTCC 3160), Gram negative microorganisms such as E.coli (MTCC 1698) and Klebsiella pneumoniae (MTCC10309) obtained from IMTECH, Chandigarh were swabbed on the surface of the agar plates. Well was prepared by using cork borer followed with loading of 50 µl and 100 µl of each sample to the distinct well with sterile distilled water as negative control and gentamycin(30mcg/disc) as positive control. The sample

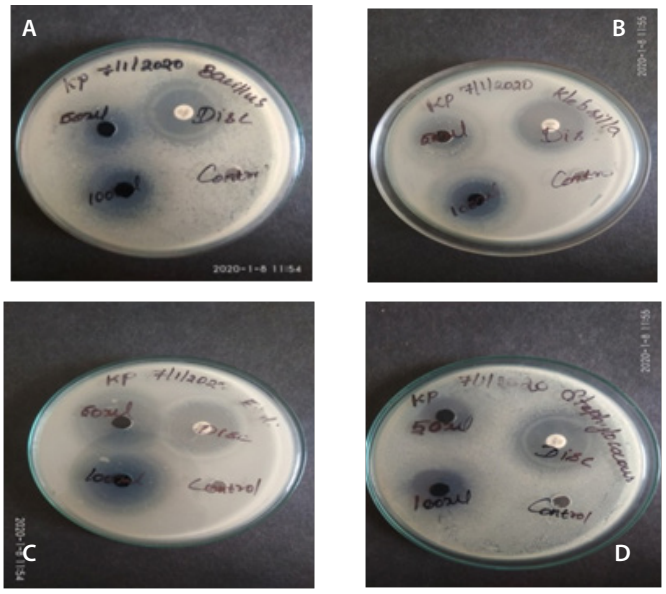

Fig. 6 (A to D): Antibacterial activity of CuS against gram positive and negative; (A) Bacteria Bacillus cereus; (B) Klebsiella pneumoniae; (C) Escherichia coli; (D) Staphylococcus aureus.

Table 2: Antibacterial activities of CuS nanoparticles

S.No	Microorganisms	Zone of inhibition in diameter (mm)			
		Control (100 µl)	Sample		Std. Antibiotic (Gentamycin) 30mcg/disc
			50 µl	100 µl	
1	Bacillus cereus	Nil	13	15	28
2	Staphylococcus aureus	Nil	15	17	29
3	Escherichia coli	Nil	25	27	27
4	Klebsiella pneumonia	Nil	18	20	27

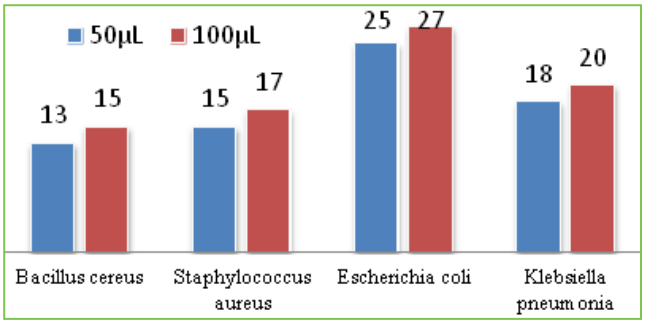

Fig. 7: Comparison graph for antibacterial activity

loaded plates were then incubated at 37°C for 24 hours to observe the zone of inhibition.

From the above graph, it is found that the diameter of inhibition zones for gram negative bacteria i.e., Escherichia coli is greater (more antibacterial effect) than other microorganisms. Thus, the copper sulphide nanoparticles exhibit an antibacterial activity over Gram positive and Gram negative organisms.

4. Conclusion

In this study, the XRD pattern reveals the crystallite size of CuS nanoparticles, depending on temperature ranging from 3-6nm. EDAX confirms the elemental composition of CuS NPs and also the spectrum indicates the presence of copper sulphide nanomaterials. The agglomeration of ball like structured nanoparticles was obtained using Scanning Electron microscope. Band gap of synthesized nanoparticles was calculated about 5.6 eV. The presence of Functional group was found out by FTIR technique. The antibacterial activity of CuS NPs has good inhibition against gram negative bacteria, Escherichia Coli. Thus, Copper Sulphide nanoparticles will open a new door for more biomedical applications and so it is inexpensive, easy and environmental friendly.

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